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Indian Standard

TEXTILE DYESTUFFS – DISPERSIBILITY OF DISPERSE DYES — DETERMINATION

UDC 677·047·42 : 677·016·8

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 2

Dyestuffs Sectional Committee, TX 17

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Dyestuffs Sectional Committee had been approved by the Textile Division Council.

Disperse dyestuffs are increasingly being used in industry for dyeing and printing fabrics. This standard has been prepared to know the dispersibility of disperse dyes and is applicable to individual dyes.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 JANUARY 2004
TO
IS 13626 : 1993 TEXTILE DYESTUFFS — DISPERSIBILITY OF DISPERSE DYES — DETERMINATION

(*Page* 1, *clause* **6.4**) — Substitute 'IS 1590 : 2000' *for* 'IS 1590 : 1960'.

(*Page* 1, *clause* **6.10**) — Substitute 'IS 2619 : 1993' *for* 'IS 2619 : 1971'.

(*Page* 1, *clause* **6.16**) — Substitute 'IS 1070 : 1992' *for* 'IS 1070 : 1977'.

(*Page* 4, *clause* **10.1**) — Insert the following Annex at the end:

ANNEX A
(*Clause* 2.1)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
878 : 1975	Graduated measuring cylinders (*first revision*)
1070 : 1992	Reagent grade water (*third revision*)
1590 : 2000	Laboratory glassware — Glass filter flasks — Specification (*first revision*)
2619 : 1993	Glass beakers (*second revision*)
5009 : 1968	Specification for Buchner funnels
6980 : 1983	Tetrasodium pyrophosphate, anhydrous, technical (*first revision*)

(TX 07)

Reprography Unit, BIS, New Delhi, India

Indian Standard

TEXTILE DYESTUFFS – DISPERSIBILITY OF DISPERSE DYES — DETERMINATION

1 SCOPE

1.1 This standard prescribes a method for determination of the dispersibility of disperse dyes as evaluated by altering time and filter residue under standard conditions in aqueous media only. Variables which affect accuracy and repeatability of the test are specified in **8**.

2 REFERENCE

2.1 The Indian Standards listed at Annex A are necessary adjuncts to this standard.

3 PRINCIPLE

3.1 A quantity of disperse dye is pre-diluted. heated and passed through filter papers of specified micron size. The time of passage and retention of dye is then evaluated.

3.2 Three tests are outlined according to intended dye application (*see* **9.6** and Table 1).

4 DEFINITIONS

4.1 Dispersibility

The degree to which particles can be broken down to some minimum size such that they will pass through the interstices of a standard filter paper.

5 SAFETY PRECAUTIONS

5.1 Good laboratory practices should be followed. Wear safety glasses in all laboratory areas and a single use dust respirator while handling powder dyes.

5.2 All chemicals should be handled with care. The compounds used in this test method at the concentrations specified are considered to present no significant hazard when used in accordance with good laboratory practices.

5.3 If concentrated acetic acid is diluted to prepare 10 percent acetic acid, use chemical goggles or face shield, impervious gloves and an impervious apron during preparation. Concentrated acids should be handled only in an adequately ventilated laboratory hood.

CAUTION : Always add acid to water.

5.4 An eyewash/safety shower should be located nearby and an organic vapour respirator with full facepiece should be readily available for emergency use.

6 APPARATUS AND REAGENTS

6.1 Buchner Funnel : Coors, glazed one-piece porcelain, 11 cm diameter (*see* **9.2**) or equivalent (*see* IS 5009 : 1968).

6.2 Filter papers : Whatman No. 2, 11 cm diameter ; Whatman No. 4, 11 cm diameter or exact equivalent filter paper from other sources (*see* **9.5**)

6.3 Stainless steel ring with approximate dimensions 103 mm inside dia, 111 mm outside dia and 8 mm height to hold filter paper in place (*see* **9.8**).

6.4 Filtering flask with side tube, capacity 1 000 ml (*see* IS 1590 : 1960).

6.5 Manometer to measure vacuum (pressure drop).

6.6 Vacuum pump or water aspirator to obtain vacuum of 56 ± 10 cm.

6.7 Vacuum control valve.

6.8 Rubber tubing vacuum.

6.9 Stop watch.

6.10 Beakers, 400 ml or larger (*see* 2619 : 1971).

6.11 Balance, analytical.

6.12 Tetrasodium salt of ethylenediamine tetra acetic acid (EDTA) as 25 percent solution.

6.13 Tetrasodium pyrophosphate (TSPP) 10 percent solution (*see* IS 6980 : 1983).

6.14 Acetic acid, 10 percent solution.

6.15 Filter residue scale (*see* **9.13**).

6.16 Distilled or deionized water (*see* IS 1070 : 1977), which does not change significantly in *p*H on heating. This water is modified with 0·25 g/l of chelating agent (EDTA) as specified in **6.12**.

6.17 Standard dye samples (*see* **9.2**, **9.3** and **9.4**).

6.18 Stirrer, laboratory, magnetic.

6.19 Graduated cylinder, 250 ml (*see* IS 878 : 1975).

6.20 *p*H meter.

7 PROCEDURE

7.1 Distilled water (*see* **6.16**) is further stabilized as follows:

Tests I and II (*For polyester dyes*) : Add 0·2 g/l of EDTA to the distilled or deionized water (*see* **7.3**).

Test III (*for nylon dyes*) : Add 0·25 g/l EDTA distilled or deionized water and adjust final *p*H (*see* **7.3**). For further explanation *see* **9.6** and Table 1.

Table 1 Selection of Applicable Test

(*Clauses* 3.2 *and* 7.1)

Test	Whatman Filter Paper Combination	Application of Dyes	*p*H of Dispersion
I	No. 2 over No. 4	Package dyeing of polyester	4·5 — 5·0
II	No. 4 over No. 4	Beck dyeing of polyester	4·5 — 5·0
III	No. 4 over No. 4	Dyeing of nylon carpet and apparel	9·0 — 10·0

7.2 Weigh out 2·0 ± 0·1 g of dye powder or 4·0 ± 0·2 g of 50 percent liquid of the dye sample to be tested. Also weigh out an equal quantity of the standard dye (*see* **9.3** and **9.4**).

7.3 Add the dye weighed in Section **7.2** completely and slowly to 200 ml of the vigorously agitated distilled water (*see* **6.16**) at 45-50°C in a 400 ml beaker. Use a magnetic stirrer. Adjust *p*H (*see* **9.15**) as follows:

*Test I and II p*H *4·5* : 5·0 with acetic acid (*see* **6.14**) ; Test III *p*H 9-10 with TSPP (*see* **6.13**, **9.10** and **9.11**).

7.4 Heat for 5 to 10 minutes at 70°C. Stir by means of a magnetic stirrer to prevent localized heating (*see* **9.1** and **9.12**).

7.5 Pour 200 to 300 ml of water at 70°C through a 11 cm funnel without filter paper (*see* **9.9**). Wait for 25 ± 10 seconds. Turn vacuum on until the water has passed through the funnel.

7.6 Cut vacuum off and immediately place filter papers in Buchner funnel (*see* **9.5**, **9.6** and **9.8** *and* Table 1).

Test I :	Whatman No. 2 on top Whatman No. 4 on bottom
Test II :	Whatman No. 4 on top Whatman No. 4 on bottom
Test III :	Whatman No. 4 on top Whatman No. 4 on bottom

7.7 Insert stainless steel ring in funnel over filter papers and turn on vacuum. Allow vacuum to reach 56 ± 10 cm (*see* **9.7**).

7.8 With vacuum on, immediately pour dye dispersion which has been heated to 70°C (*see* **7.4**) into funnel and begin timing. Record time up to 120 seconds to the nearest second. The end point is reached when the appearance of the filter paper changes from a wet to a dry look.

7.9 Allow filter paper to dry and evaluate as outlined in 7 (*see also* **9.14**).

8 EVALUATION

8.1 Record time for filtration and rate as follows:

Class A – 0 – 24 sec
Class B – 25 – 49 sec
Class C – 50 – 74 sec
Class D – 75 – 120 sec
Class E – > 120 sec

8.2 Examine residue on filter paper *versus* the Filter Residue Scale (standard photographic replicas) (*see* **9.13**). Also examine the paper for any visible coarse or granular particles (*see* **9.13**). If present, the test is automatically rated as Class I. If non is present, rate as follows:

Class 5 — Excellent
Class 4 — Very good
Class 3 — Good
Class 2 — Average
Class 1 — Poor

9 PRECISION ON BIAS

9.0 Precision and bias for this test method are being established.

9.1 The results of this test method can vary widely unless all details are run under the conditions outlined. Any variation in the conditions can cause the test to be invalid. Result have been shown to be repeatable in several laboratories when the specified conditions were met.

9.2 Variations in results can be caused by differences in the diameter of the filter funnel due to differences in surface area.

9.3 The dye standard as agreed between the buyer and the seller is always included so that the influence of any operator induced variable on the results is minimized.

9.4 When evaluating two different dyes of approximately equal shade but of different tinctorial value, one dye is always selected as standard. Then the other product is run on an equal strength (grams/200 ml) basis using as a guide the Standard Depths dyeings (18 hues) as available from AATCC. When evaluating two samples of the same dye, the standard is the one furnished as standard material by the respective dye manufacturer. When evaluating the same dye from two different manufacturers, one should determine if the two products should be evaluated as outlined in the first two sentences of this paragraph.

9.5 Whatman paper is specified because it was used by all participants in developing this test method. Any other filter paper which gives equivalent results may be used. Two filter papers are used to provide a more uniform vacuum across the filtering surface. The filter paper is used as received in the box with the slick side up to avoid variations due to texture differences on the rough side. The coarser paper should always be on the bottom. Other papers may be used if quality and micron retention are exactly the same as the Whatman paper.

Whatman No. 2 — Filters particles above 8 μm.

Whatman No. 4 — Filters particles above 25 μm.

9.6 Tests I, II and III are defined as follows:

Test I — Whatman No. 2 filter paper over Whatman No. 4 filter paper. This test is designed to evaluate the most critical dispersion requirements such as for package dyeing of polyester.

Test II — Whatman No. 4 filter paper over Whatman No. 4 filter paper. This test is designed to evaluate dispersion requirement for dyeing at longer liquor ratios (such as beck dyeing).

Test III — Whatman No. 4 filter paper over Whatman No. 4 filter paper. This test is designed to evaluate the dispersion requirement of disperse dyes for application on nylon (carpet and apparel dyeing). The *p*H of 9 – 10 specified for this test reflects common industrial practice.

9.7 The vacuum should be 56 ± 10 cm. This range is obtained by using either a standard vacuum pump or house vacuum. However, a water respirator may not give this vacuum untill a liquid is introduced into the system. In this event the test should proceed after pouring the dye into the funnel and waiting 10 – 13 seconds for full vacuum.

9.8 Since the Whatman filter paper is cellulosic in nature, it can swell quite readily when wet out. Therefore, if the paper is prewet, the time of filtration will go up as a result of the wet out temperature and/or time lag. Thus it is imperative that the paper should not be wet prior to the test, since the micron retention of the paper is reduced. This is the reason for the stainless steel ring, which holds the filter paper in place without prewetting. On dyes which have a very small particle size, the speed of filteration will not be changed to a sizable degree by a vacuum alteration. However, a reduced vacuum can result in faster filtration times on dyes of larger particle size, since the suction is not as large. On dyes of larger particle size, an increased vacuum will block the paper faster, slowing up the filtration rate, and thus swelling the paper more. This results in higher filtration times and more residual dye on the paper.

9.9 The funnel should always be preheated with 200-300 ml of water heated to 70°C and then wiped dry so that the dispersion will not be reduced in temperature by a cold funnel. The preheating cycle should always come just prior to running the tests without the paper in place.

9.10 A concentration of 2 g/200 ml was chosen as representing the upper limit of normal dye application. For dyes of high tinctorial strength, this can be reduced to 1 g/200 ml, but it should always be reported that 1 g was used.

9.11 It is very important to use 45-50°C water. Wide variations can result with certain dyes if water at 70°C is used in the beginning. The specified procedure allows the dye to wet out under conditions similar to those used in plant practice.

9.12 Excessive time lags and holding temperatures may affect the outcome of the test. Test should be completed within 15 minutes.

9.13 The Filter Residue Scale consists of photographs representing five classes of retention of filter residue and is used as part of the evaluation procedure (*see* **8.2**). Available from AATCC. P.O. BOX 2215, Research Triangle Park NC 27709, USA.

9.14 In order to give more reproducible results, especially with dyes in Class 3 or lower, it may be advisable to rinse the filter paper while it is in the funnel with 10-15 ml of specially prepared water before drying (*see* **6.16**). This will aid in the removal of any colloidal dye that actually has a micron size smaller than the paper being employed.

9.15 It is necessary to adjust the *p*H at this point since various dyes have wide *p*H ranges themselves. For example, the *p*H of dispersions of paste dyes usually is more alkaline than of dispersions of the same dyes in powder form.

10 REPORT

10.1 The test report shall include the following:

a) Test Number (*see* **9.6** *and* Table 1).

b) Class according to filtration rate (*see* **8.1**)

c) Class according to amount of residue (*see* **8.2**).

Example : 1–A–3 where 1 the test Number (Whatman No. 2 over Whatman No. 4) ; A indicates the time of filtration (0–24 sec.) ; and 3 represents amount of residue on filter paper as compared with a standard photographic chart.

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